

NBSIR 73-231

Response Characteristics of a Portable X-ray Fluorescence Lead Detector: Detection of Lead in Paint

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Center for Building Technology
Institute for Applied Technology
National Bureau of Standards
Washington, D. C. 20234

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Final Report

Prepared for
Office of Policy Development and Research
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U. S. DEPARTMENT OF COMMERCE, Frederick B. Dent, Secretary

NATIONAL BUREAU OF STANDARDS, Richard W. Roberts, Director

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Abstract

The objective of this investigation was to obtain an indication of the validity of the field data resulting from the use of a portable x-ray fluorescence lead detector commonly used by local lead paint detection programs. This report is intended to provide guidance in the use of portable x-ray fluorescence lead detectors by housing and/or health authorities who are responsible for the collection and interpretation of field data as part of lead paint control programs.

The response characteristics of such an instrument to conditions that are related to those encountered in the field have been investigated and the results are presented in this report. The affects of calibration standards, state of charge, paint overlayers, substrate, and distance on instrument response are discussed, in addition to the limit of detection and precision. The accomplishment of these tasks required the development of panel-type lead calibration standards. These standards encompass the concentration range from 0.1 mg/cm^2 to 9.0 mg/cm^2 .

Key Words: Leaded paint detection; portable x-ray fluorescence lead detector; portable x-ray fluorescence lead calibration standards.

Response Characteristics of A Portable X-Ray Fluorescence
Lead Detector: Detection of Lead in Paint

1. Introduction

The Princeton Gamma Tech Model XK-2* is a portable x-ray fluorescence (XRF) instrument which detects the K x-ray emissions of lead. The class of instruments this particular model represents is presently thought to have the most potential for application in lead paint detection programs. However, a major drawback to the proper use of these instruments in the field has been the lack of a test program which adequately demonstrated their response under various field conditions. The response characteristics of an instrument must be documented before it can be used with any assurance of obtaining correct results. Although Graveson, et al [1] and Rasberry [2] have studied a number of portable XRF lead detectors, a more thorough investigation of the Model XK-2 has been conducted because of its wide use in local lead paint programs.

The response characteristics of this instrument have been investigated under laboratory conditions in order to identify and quantitate the affects of various simulated field conditions on instrument response. Such a study was necessary in order to ascertain the validity of the field data resulting from the use of this instrument. Although the

* In no case does such identification imply recommendation or endorsement by the National Bureau of Standards, nor does it imply that this instrument is necessarily the best available for the purpose.

primary objective of this study was to define the response of these instruments to situations that could be expected to occur in the field, experimental work was done in the laboratory in order to obtain clearly defined, reproducible conditions. Some caution is therefore warranted when attempting to extrapolate the findings of this study to actual field conditions. This study is simply a guide to the type of response that can be expected under various conditions and should not be used in a totally quantitative manner.

Panel-type calibration standards of accurately known lead content were not available when this study was commenced. This made it impossible to determine the response characteristics of the Model XK-2 in the concentration range of interest to most lead paint detection programs; between 0 and 2 mg/cm². Therefore, a secondary objective was the development of calibration standards for portable XRF lead detectors.* Eight standards have been made by a vacuum deposition of pure lead onto glass plates. The concentration range of these standards is from 0.10 to 9.0 mg/cm².

The 10 instruments used in this study were purchased by the Department of Housing and Urban Development (HUD) for use in lead paint detection programs in various cities. They were purchased as new instruments but some were taken into the field by NBS personnel during

* The use of the work "standard" is not meant to imply that these plates have been certified by NBS. It simply means that the concentration and homogeneity of the lead film is known with sufficient accuracy for them to be used as a primary reference.

the progress of this study for use in a housing survey. Therefore, some of the instruments were subjected to field use before the study was completed. This expanded the scope of the study to the testing of used as well as new instruments. Suggestions originating from the personnel involved in the field use of the instruments were incorporated into the investigation.

A brief study was also made of the response of a Chicago-Nuclear Model 9257* portable XRF lead detector to the lead calibration standards. This instrument, which is the old version of the Model Pb-3, exhibited a negative bias in its calibration curve (as did the Model XK-2). A response of -1.2 mg/cm^2 was obtained at 0 mg/cm^2 and a response of 7.9 mg/cm^2 was obtained at 9.0 mg/cm^2 . The fact that both models of the portable XRF lead detector exhibited systematic errors, even though the instruments employ different detector systems, suggests that possibly all portable XRF instruments should be calibrated before they are used in the field.

2. Summary

The response of a portable XRF lead detector to our lead calibration standards of accurately known concentration was investigated under laboratory conditions. The objective was to define the response of the

instruments under simulated field conditions, thereby aiding in the interpretation of field data. The results of the study are as follows:

1. The calibration verniers on the front panel of the instrument require periodic adjustment. The peak vernier requires re-setting about once every two weeks, the calibrate vernier at least once a day, and the zero vernier every 250 readings.
2. The calibration procedure outlined by the manufacturer is really only a standardization procedure and not a true calibration. Field data should be referred to a calibration plot in order to obtain a corrected reading.
3. The limit of detection for a single reading is approximately 0.4 mg/cm^2 . This corresponds to roughly one layer of 6% lead in paint or six layers of 1% lead in paint.
4. Assuming the same quantity of lead is present, lower readings will be obtained on metal substrates (window casings, cabinet doors, etc.) than on plaster or wood substrates. A separate calibration plot should be used for metal surfaces.
5. A minimum of 300-400 consecutive readings can be expected from a single overnight charge.
6. The range for a single reading is $\pm 1 \text{ mg/cm}^2$, while the 95% confidence interval for an average of 4 readings is $\pm 0.5 \text{ mg/cm}^2$.
7. Paint overlayers, even those containing ZnO , do not seriously affect the readings obtained for lead concentrations of approximately 1 mg/cm^2 or lower.

8. A separation of 1.9 cm between the detector and the surface that is being measured will cause a reduction in the results of approximately 90%. This effect is important for odd-shaped surfaces.

3. Experimental

3.1. Preparation of Lead Calibration Standards

The lack of panel-type lead standards for use in the calibration of portable XRF instruments has been a major obstacle to the effective use of these instruments in the field. Since x-ray methods of analysis are not absolute, but relative, the accuracy of an analysis is largely dependent upon the accuracy of the calibration procedure. Therefore, the more closely the calibration standard resembles the material to be analyzed, the greater the accuracy that can be expected for the analysis. This suggests that the ideal standard would be a wooden panel (representing a typical wall substrate) covered with a paint film of known lead content in terms of milligrams per square centimeter (mg/cm^2). However, a dry paint film of adequate homogeneity is very difficult to produce, and readings tend to vary by as much as 50% from point to point on such films. Therefore, paint films are not acceptable as calibration standards.

Since a painted-panel standard was not practical, a pure lead panel-type standard was investigated. The lead plate standards were prepared by a vacuum deposition of ACS reagent grade lead pellets on 7.62 cm x 7.62 cm x 0.48 cm glass plates [3]. Glass was chosen as the

substrate because of its mechanical strength, transparency to x-rays, and uniform surface. The density of lead was taken to be 11.344 g/cm^3 (20°C) and the thickness of the deposited lead film was measured interferometrically. The density, in addition to the film thickness, allowed the calculation of the weight of lead per unit area. Lead-coated plates were prepared with values of 0.1, 0.2, 0.5, 1.0, 1.9, 3.0, 4.6, and 9.0 mg/cm^2 .

Since the lead films were very delicate, the plates were sprayed with acrylic lacquer to protect the lead from abrasion.

The inhomogeneity of the film thickness was calculated to be approximately $\pm 2\%$, which is the uncertainty in the accuracy of the lead films between 0.1 and 4.6 mg/cm^2 [3]. The accuracy of these plates is sufficient to make them acceptable as standards for calibrating portable XRF instruments. The lead film of 9.0 mg/cm^2 was too thick to be measured interferometrically. This plate was weighed before and after the lead was deposited and the weight of the lead was divided by the surface area of the glass plate. The accuracy of this plate, since the error in the measurement of the surface area was less than $\pm 1\%$, should still be within $\pm 5\%$ of the nominal value.

3.2. Preparation of Free Paint Films

Non-leaded paint films were prepared and used to simulate over-layers of paint. This was done in conjunction with the standard lead plates. The films were prepared by drawing down a paint layer on photographic paper, allowing the paint to dry, and peeling it off to yield a flexible, free paint film [4]. The paint films were then sandwiched between thin plastic sheets for protection.

The compositions of the paint films are described in table 1. Two formulations were used in making the paint. However, the important difference in the formulas is the inclusion of 7.2% zinc oxide (ZnO) in the second formulation. Thirteen paint films were produced which did not contain ZnO, and seven were made which did contain ZnO. The average thickness of the paint films were 2.9 and 3.3 mils, respectively, as measured by a micrometer.

3.3. Statistical Treatment of Data

The terms accuracy and precision are often confused when discussing an analytical procedure, since they concern different aspects of the same problem. Accuracy is the agreement between the observed value for an analysis and the true value. The accuracy of x-ray analytical methods is determined by the adequacy of the calibration standards that are available and the ability to control as many experimental variables as possible [5]. Precision refers to the ability to consistently duplicate the observed value when an analysis is repeated, regardless of the true value. Therefore, the accuracy and precision of an analysis are independent of each other. The precision of x-ray methods is based upon statistical averaging; the more data that is available, the better the precision of the analysis. This can be discussed in terms of a normal distribution curve, which is illustrated in figure 1 [6].

A normal distribution curve can be used to describe the precision as long as the observed values from a number of measurements have an equal chance of being greater or less than the mean value. This is valid for x-ray methods [5]. The precision of an analysis is determined by

Table 1

Composition of Paint Films

Containing No Zinc Oxide

TiO ₂	2000 g
Silicates	1300 g
Vehicles	6700 g

Nonvolatiles = 55.0%

Containing Zinc Oxide

TiO ₂	260 g
CaCO ₃	40 g
ZnO	40 g
Vehicle	660 g

Nonvolatiles = 55.5%

7.2% ZnO by weight in nonvolatiles

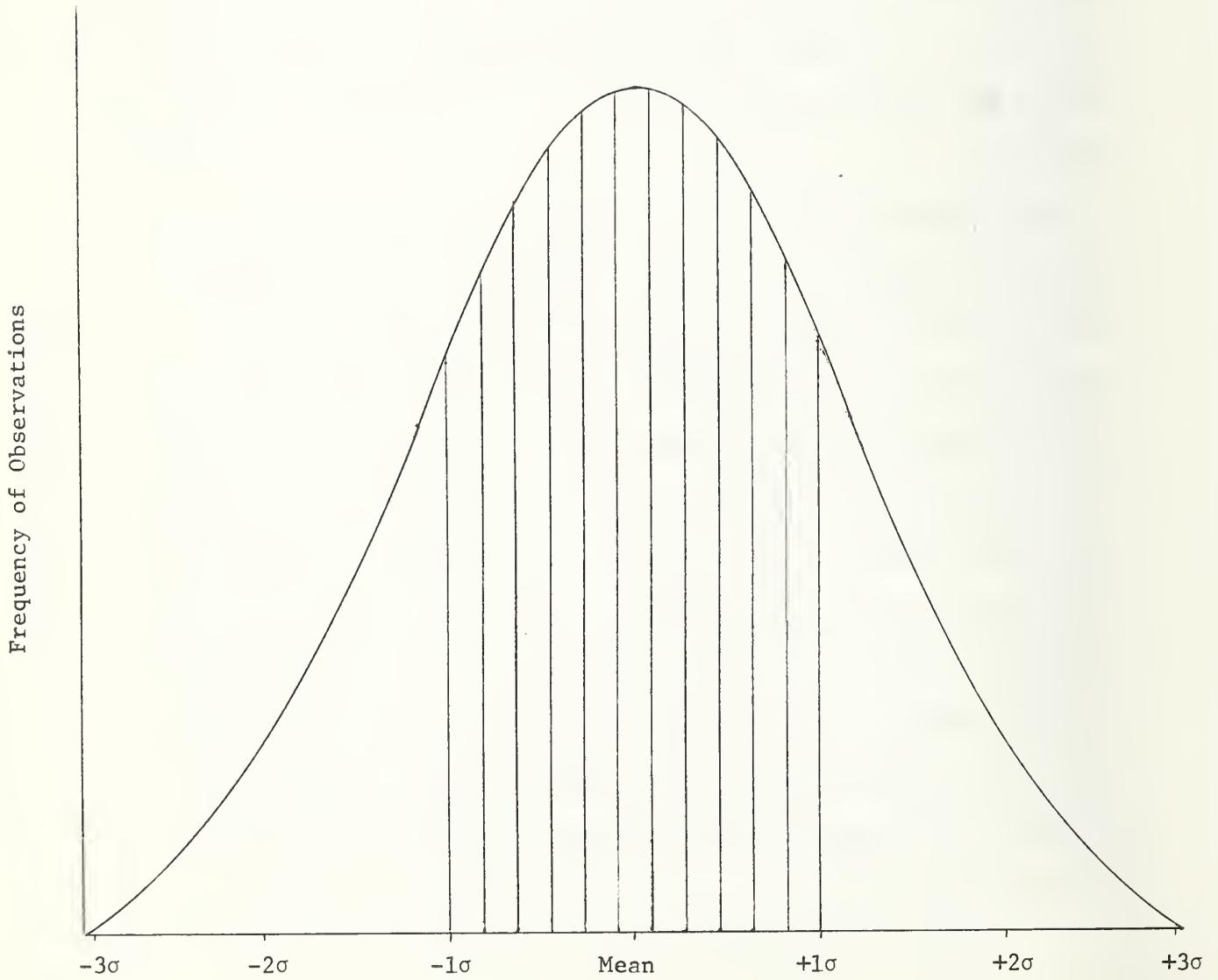
the spread of the curve about the central value; the narrower the curve, the better the precision. Since this spread is measured by the standard deviation (σ), the smaller the standard deviation, the more precise the analysis.

The cross-hatched area in figure 1 represents a deviation of $\pm 1\sigma$ from the central value. This area encompasses 67% of the total area under the curve. This means that an observed value will be within $\pm 1\sigma$ of the central value 67% of the time. In a similar manner, the observed value will be within $\pm 2\sigma$ of the central value 95% of the time, and within $\pm 3\sigma$ 99.7% of the time. The standard deviation is determined experimentally and reported in the same units as the measurement, in this case mg/cm^2 [6].

The standard deviation for an analysis by x-ray methods is proportional to \sqrt{N} , where N is the number of counts, or x-ray emissions, observed by the detector [5]. Therefore, the more counts that are collected, the greater the precision of the analysis. Since the count rate is constant on a time-averaged basis, the number of counts which will be collected for a given concentration of lead is directly proportional to the length of time that counts are collected. Portable XRF lead detectors, such as the Model XK-2, employ count times of approximately 10-15 seconds per reading. This means that the standard deviation of the mean for an average of 10 readings should be only about a third of the standard deviation for a single reading. All the data contained in this report, unless specified otherwise, are an average of 10 readings.

Figure 1

Normal Distribution Curve



4. Results

4.1. Calibration

4.1.1. Instrument Standardization

The XRF instrument is standardized by following the calibration procedure outlined by the manufacturer [7]. Each instrument is supplied with 2 calibration standards: a plain wooden block for calibrating at 0 mg/cm^2 and a wooden block covered with lead foil for peaking the sensitivity and calibrating at approximately 75 mg/cm^2 . However, it must be emphasized that this procedure only results in the standardization of instrument response and is not a true calibration. It allows readings obtained at various times and from different instruments to be compared, but it does not give sufficient information for the quantitative interpretation of readings obtained in the field. This point is discussed in section 4.1.2.

Typical standardization responses are illustrated in figures 2-4. The decision as to whether or not an instrument is functioning properly can sometimes be made from plots of this type. In particular, the instrument should be capable of being zeroed; that is, negative readings should be obtainable. In addition, the zero and calibrate plots should be linear. The data necessary to construct the plots illustrated in figures 2-4 were obtained by taking 10 readings for each point on the graph and plotting the algebraic average of the 10 readings. The algebraic average is obtained by subtracting the negative readings from the positive readings and dividing the resultant sum by the total number of readings for that data point.

Figure 2

Peak Calibration

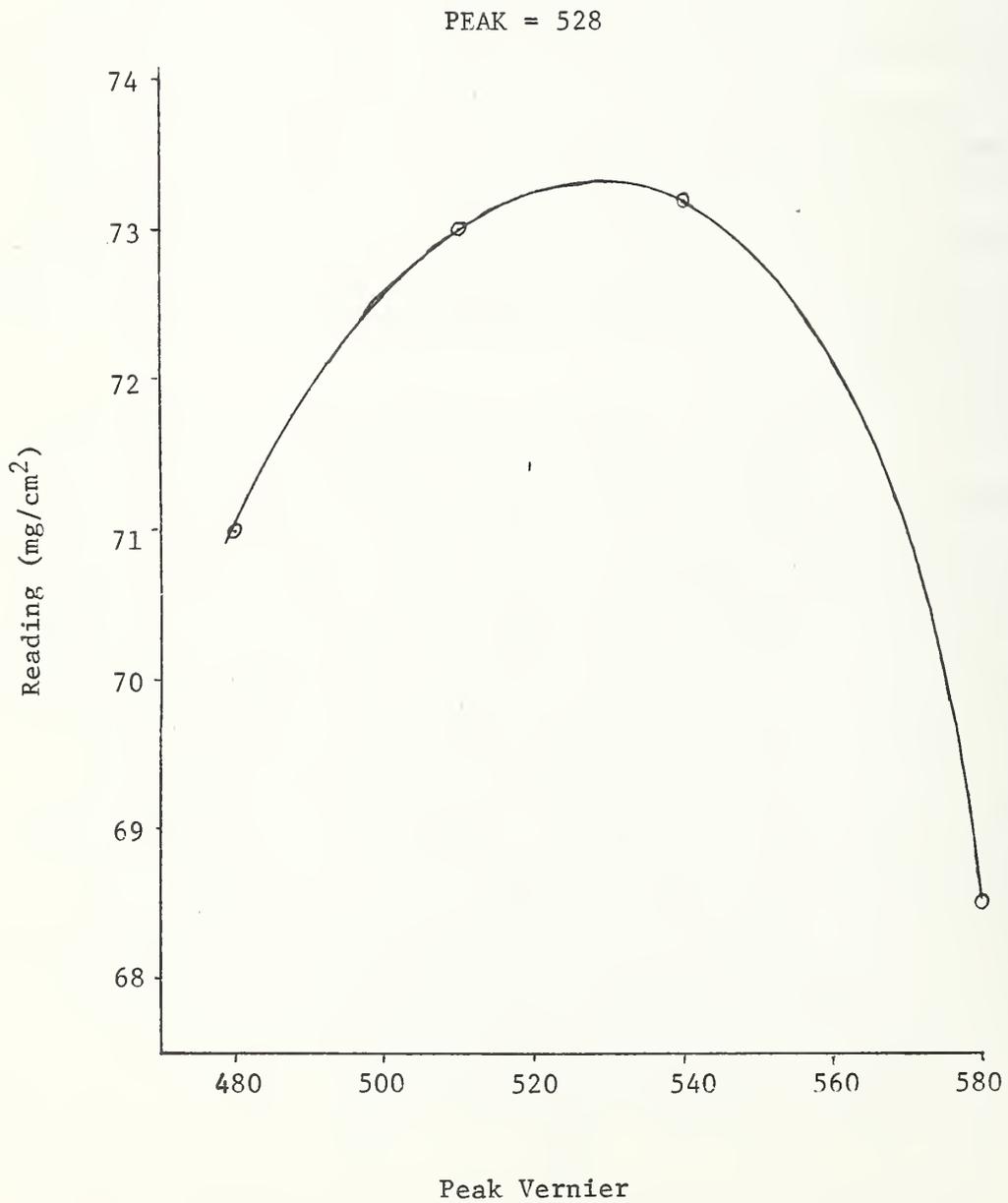


Figure 5
Zero Calibration

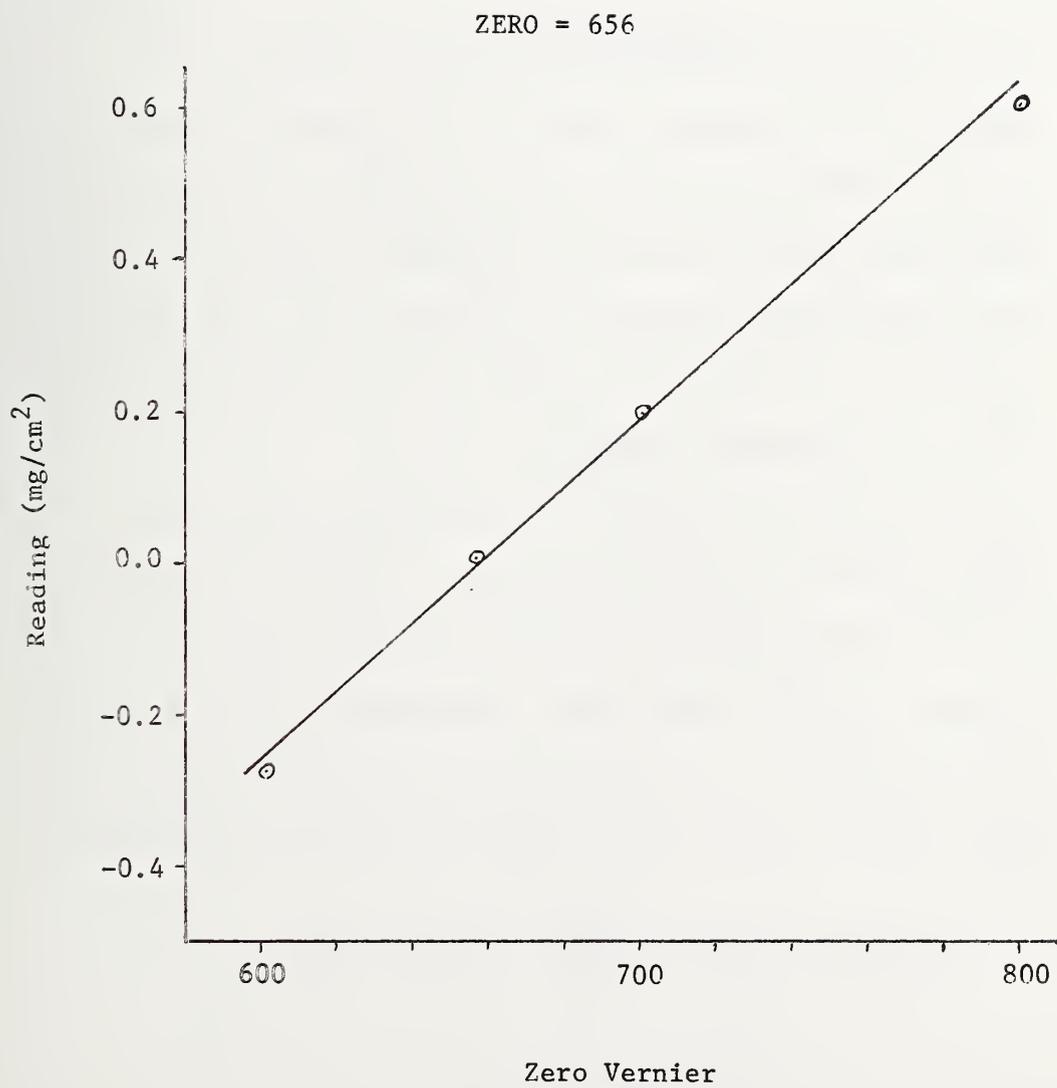
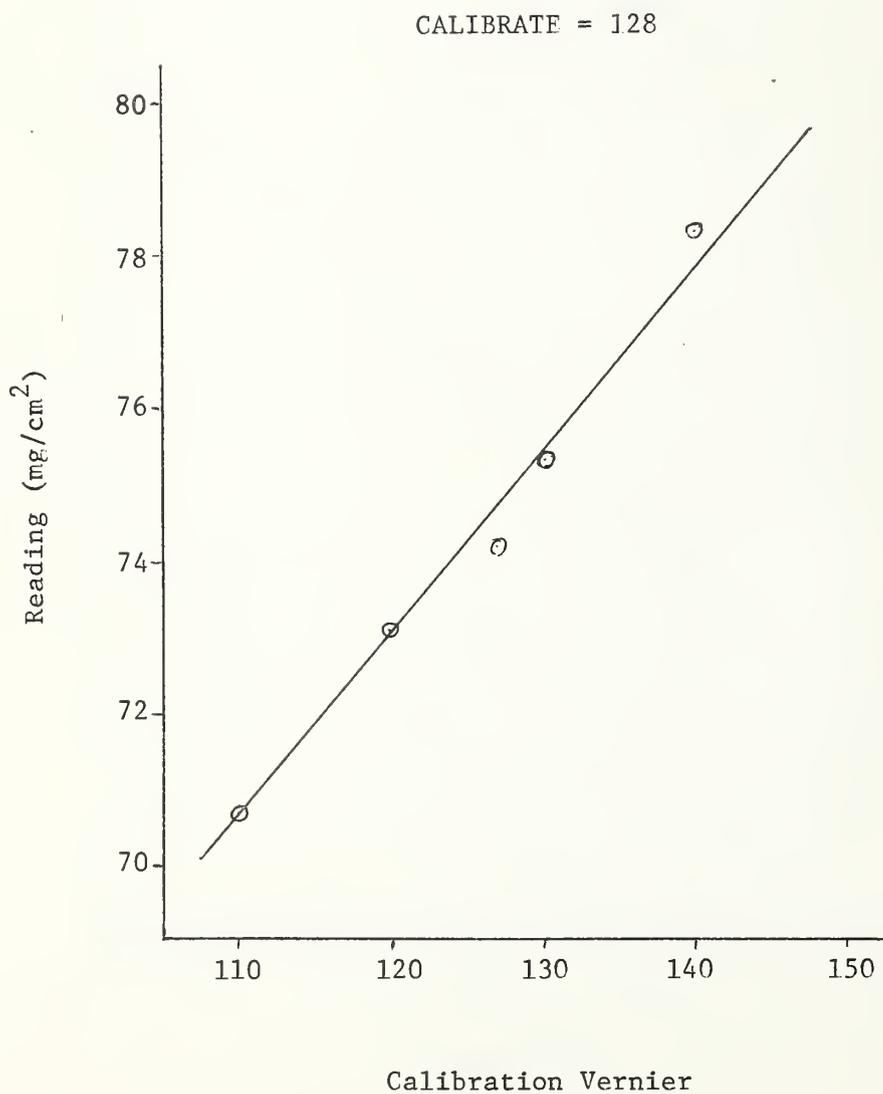


Figure 4
Calibration at 75 mg/cm²



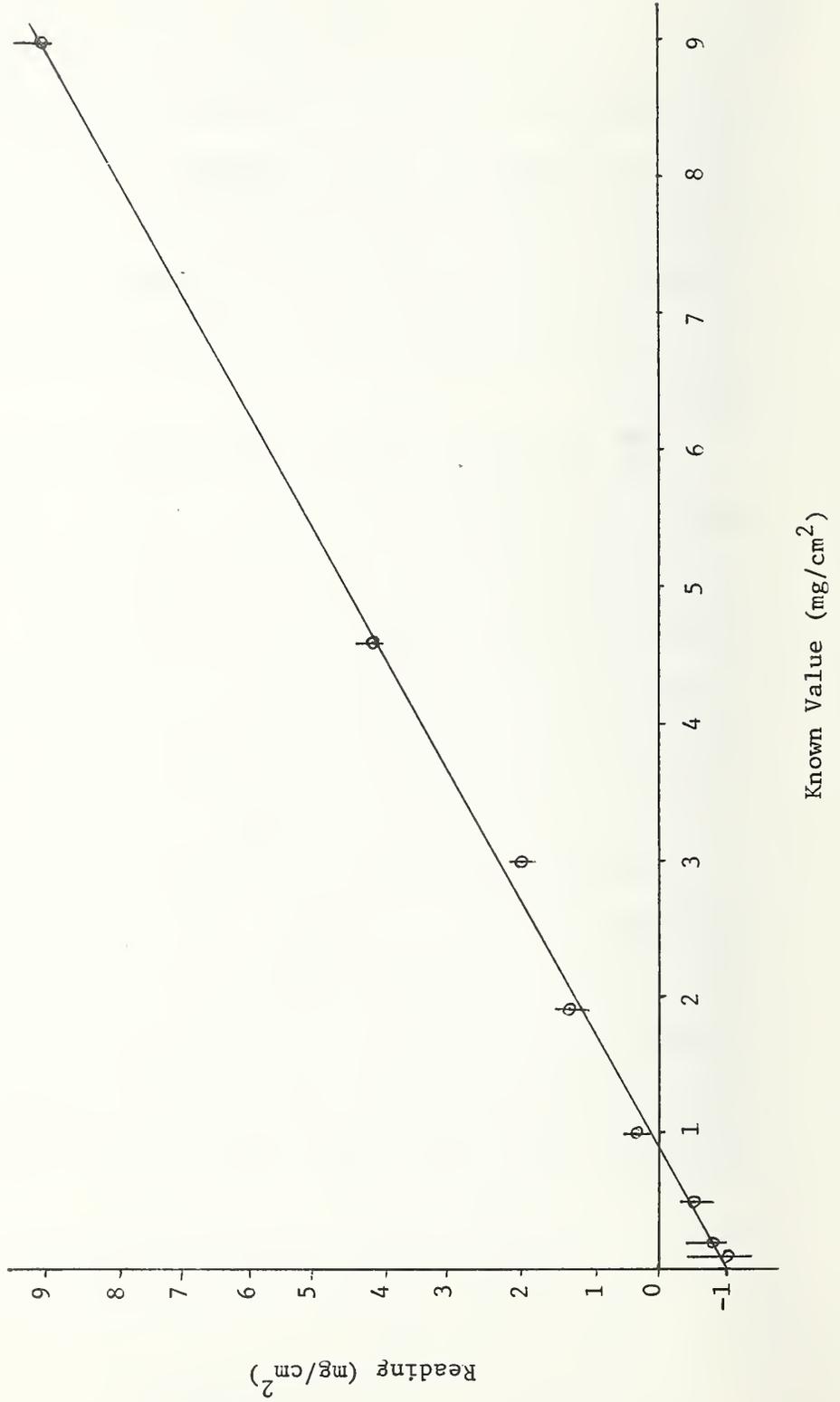
Readings were obtained by placing the lead plate standards on a sample holder made of plexiglass. The sample holder was designed so that replicate series of readings would be as reproducible as possible. The effect of the sample holder and the glass substrate used for the lead standards on readings was found to be negligible. This was determined by comparing replicate readings; the first reading was on a wooden block, and the second reading was for the sample holder (with a piece of clean glass plate) on the wooden block. The differences in the readings were either zero or within the standard deviation of the instrument.

Since instrument response is not constant with time, the instrument must be standardized at regular intervals. This corrects the data obtained at various times for instrument drift. In general, the results of this study suggest that the peak vernier should be adjusted at least once every 2 weeks, while the zero and calibrate verniers should be adjusted at least once a day while the instrument is in use.

4.1.2. Instrument Calibration

The average response of 8 instruments to the lead plate standards is depicted in figure 5. The peak, zero, and calibrate verniers were set at the proper values by following the standardization procedure outlined by the manufacturer. Ten readings were then taken for each NBS lead plate standard and the average and standard deviation were calculated for each set of data. This process was then repeated for

Figure 5
Average Response of Eight Instruments



the 8 values obtained at each lead standard, so that the data points plotted in figure 5 are an average of 80 individual readings. The range of response for the 8 instruments is indicated for each data point.

Figure 5, which is a true calibration curve, suggests that (a) negative readings are valid and should not be recorded as zero, (b) instrument response versus concentration is linear between 0.1 and 9 mg/cm², (c) the readings obtained in the field must be compared to a calibration curve in order to arrive at a true lead content, and (d) the response from different instruments, in different locations, can be compared with a reasonable degree of reliability if all the instruments have been properly standardized.

The observed negative bias in the calibration curve seemed to be caused by a combination of effects. The most pronounced shift in the calibration curve was obtained by peaking the instrument at 9.0 mg/cm² rather than at 75 mg/cm² as suggested by the instruction manual [8]. The instrument was then zeroed on a wooden block and calibrated with the 9.0 mg/cm² standard. Following this procedure resulted in a linear calibration curve between -0.4 mg/cm² at a value of 0 mg/cm² and 8.9 mg/cm² at a value of 9.0 mg/cm². This procedure corrected for approximately half the negative bias.

The remainder of the negative bias was removed by essentially following the above procedure (peaking at 9.0 mg/cm²), but not using the wooden block to zero the instrument. Instead, the instrument was

adjusted, using the zero vernier, to read 0.1 mg/cm^2 for the calibration standard with that value. This procedure yielded a linear calibration curve between 0 and 9.0 mg/cm^2 . The negative bias can be eliminated, to a good approximation, by peaking and calibrating the instrument with the lead foil supplied by the manufacturer and adjusting the zero vernier to obtain an average reading of 0.4 mg/cm^2 on the wooden block.

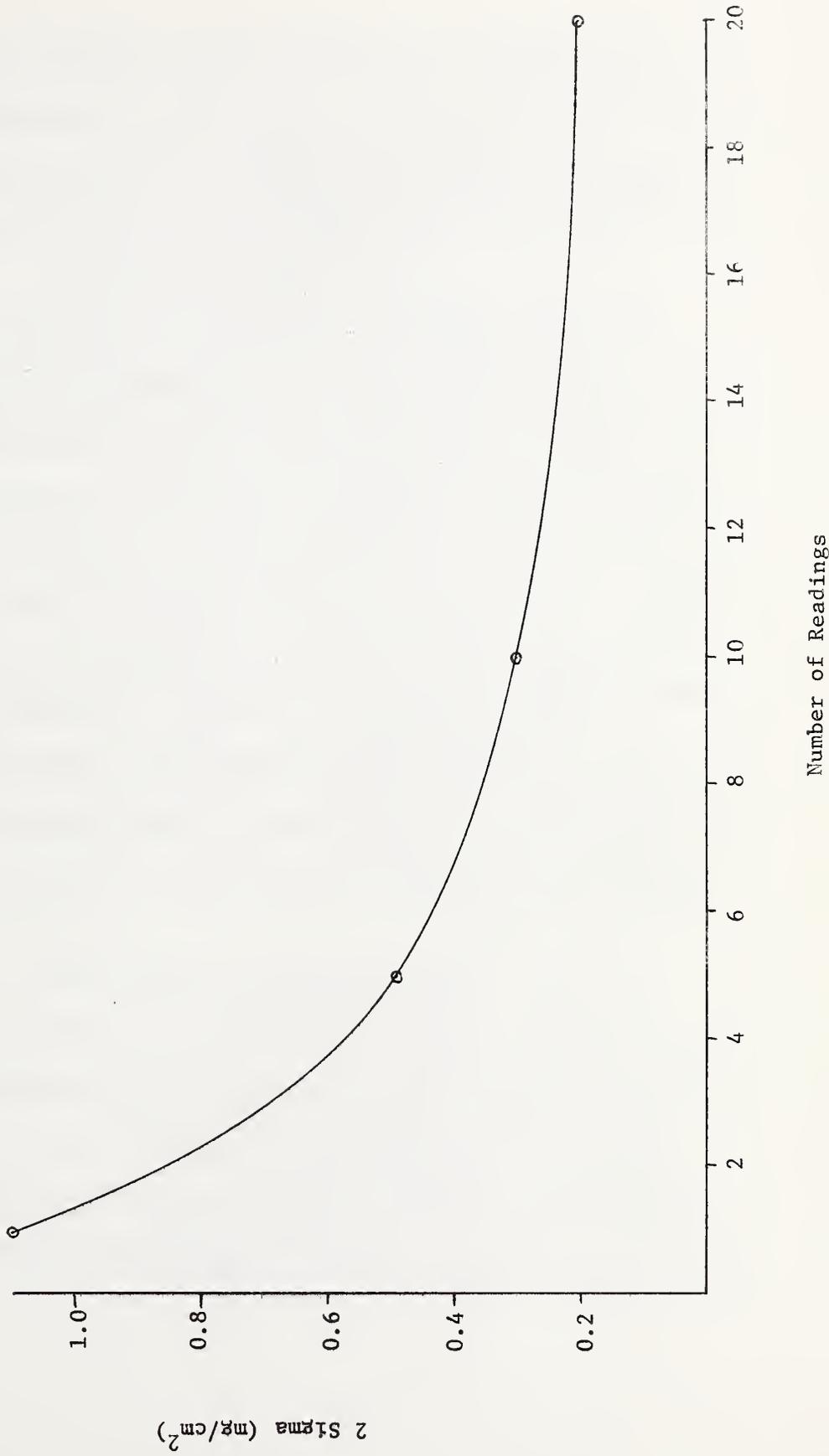
4.1.3. Precision

Although the calculated standard deviation is a measure of the variability that is expected for a reading, the data in figure 6 is presented in terms of 2σ , where σ refers to the standard deviation of the mean. This represents a 95% confidence interval; that is, only 5 of every 100 readings would be expected to vary from the mean by more than the values shown in this figure. The data in figure 6 represents the averaged response of 2 instruments, and twenty readings were taken with each instrument on the lead plate standards. Standard deviations were calculated for each lead standard and these were averaged to yield an overall σ for each instrument. This approach seemed to be valid since σ was independent of lead concentration between 0.1 mg/cm^2 and 9.0 mg/cm^2 .

Figure 6 suggests that the precision of a single reading, under ideal conditions, is approximately $\pm 1 \text{ mg/cm}^2$. The precision is increased to approximately $\pm 0.3 \text{ mg/cm}^2$ for an average of 10 readings, but it does not improve appreciably with more readings. Although the precision of the reading must be balanced against the available time when collecting field data, the averaging of at least 4 readings should be

Figure 6

Variation of Two Sigma with the Number of Readings



considered since the precision indicated in figure 6 is probably the maximum that can be expected. Such factors as the matrix effect, unknown substrates, and the inhomogeneity of painted surfaces will tend to cause the value of 2σ to increase for actual field data.

4.1.4. Limit of Detection

The limit of detection is defined in this report to have the same value as the standard deviation, and represents an uncertainty of 100% in the indicated lead content of the sample. This corresponds to the sensitivity of the instrument as defined by the manufacturer. Although the limit of detection generally refers to a single reading, it can be improved by taking the average of a number of individual readings. The standard deviation for an average is calculated by dividing σ for a single reading by \sqrt{n} , where n is the number of readings. Therefore, an average of 10 readings should be approximately 3-fold more precise than a single reading. The precision in the case of an average refers to the ability of the instrument to duplicate the mean value with a replicate series of readings.

The average standard deviation for 8 instruments is 0.43 mg/cm^2 , and, as defined, this value corresponds to the limit of detection for a single reading. However, the limit of detection for an average of 10 readings would be approximately 0.13 mg/cm^2 .

The limit of detection can also be expressed in terms of a percent-layer [2]. This can be calculated by assuming an average value for the density and the thickness of an aged-paint film. If the density of a

typical paint film were assumed to be 1.0 g/cm^3 , and the thickness were assumed to be 2.5 mils, the weight of the paint film would be 6.4 mg/cm^2 [4]. A concentration of 1% lead in the paint solids would then be equivalent to 0.064 mg/cm^2 of lead in the paint film. Since the limit of detection is 0.43 mg/cm^2 , the limit of detection in terms of percent-layers is approximately 6. One layer of 6% leaded paint, or 2 layers of 3% leaded paint, or 6 layers of 1% leaded paint would all correspond to the detection limit of 0.43 mg/cm^2 of lead. The limit of detection for an average of 10 readings, because of a reduced standard deviation, would be roughly 2 percent-layers.

4.2. Factors Affecting Instrument Response

The method of x-ray fluorescence analysis is by now a standard technique in many laboratories. Therefore, a detailed description of the method is not warranted. A discussion of the factors that can be expected to influence the response of portable XRF instruments, however, is pertinent to the objectives of this report. A number of these factors are discussed in this section.

4.2.1. Effect of Battery Charge

This instrument operates on a Ni-Cd battery pack with a built-in recharger. A fully charged battery should be capable of operating the instrument, with the trigger taped open, for approximately 5 hours [8]. Assuming an average reading time of 20 seconds, a maximum of 900 readings should be obtainable from an instrument that has been charged overnight.

No more than 400 consecutive readings were ever attempted during this study, however, and the actual number of readings that can be obtained from a single charge is not known.

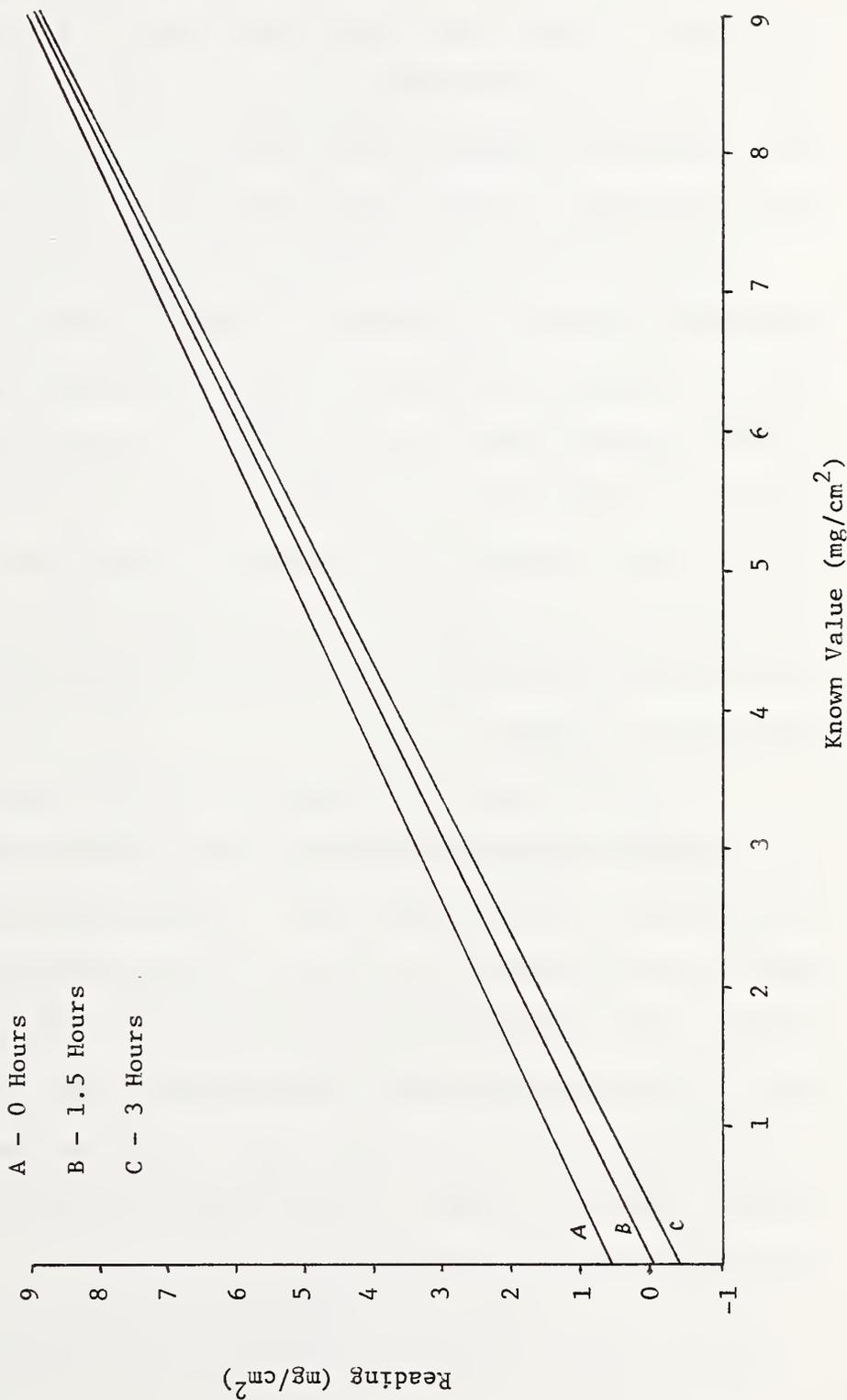
The effect the state of charge has on instrument response is illustrated in figure 7. The data refer to an instrument which had been fully charged overnight, but not standardized. The instrument was then discharged in one hour steps by taping the trigger open and taking readings at the end of each hour. This was done in order to observe any variation in response due to the state of charge. As indicated in figure 7, there is an apparent drift in instrument zero as the battery pack is discharged. These plots indicate that the zero vernier should be adjusted after an hour of continuous operation. The plot labeled 1.5 hours is the average of the data for discharge times of one and two hours; this was done to maintain clarity in the figure. Since this particular instrument had a count time of about 15 seconds, a three hour discharge represented approximately 720 readings.

4.2.2. Paint Overlayers

Non-leaded layers of paint which cover the leaded paint, whether or not they contain ZnO, will tend to cause low readings. This occurs since even elements of low atomic number, such as titanium and calcium, scatter the K x-rays of lead to some degree. However, overlayers of paint which contain ZnO will cause a greater effect since zinc absorbs the lead K x-rays.

Figure 7

Effect of Charge on Instrument Response



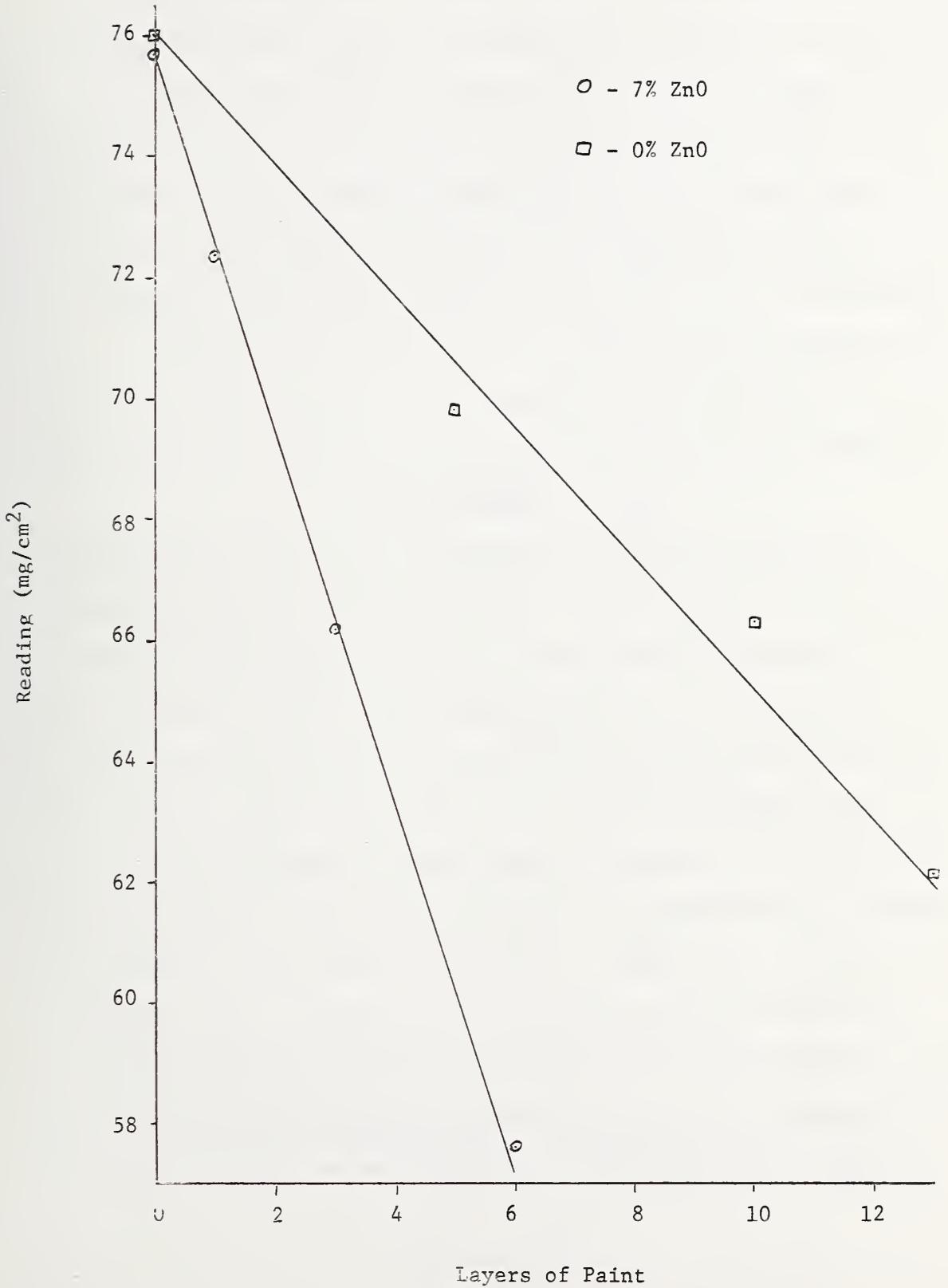
The concept of percent-layers has been applied to the use of the ZnO-containing paint films. That is, two layers of 7% ZnO were used to simulate a single layer of paint containing 14% ZnO, and four layers were used to simulate a single layer containing 28% ZnO. This can be done even though percentages are not additive, since the absorption of the lead K x-rays is proportional to the weight per unit area of ZnO.

If the assumption were made that lead-based paints have not been extensively used for the interiors of houses for over 20 years, it would be reasonable to expect any interior lead-based paint to be covered with more recently applied layers of non-lead paint. If the additional assumptions are made that an interior surface was painted every 3 years, and each time it was given 2 coats of paint, the number of overlying non-lead layers of paint would be 14. The affect of such overlying paint layers on the response from this instrument is illustrated in figure 8.

The data in figure 8 were obtained by covering one of the lead foil standards supplied by the manufacturer with increasing numbers of the previously described paint films. Although both paints caused a reduction in the reading, the affect of the zinc-based paint was much greater. The paint with 0% ZnO caused a 14% reduction in the reading after 10 layers had been added, while the paint with 7% ZnO caused a 13% reduction in the reading after only 3 layers had been added. However, the data in figure 7 tend to overstate the problem associated with the collection of field data.

Figure 8

Attenuation of Response From Paint Overlayers



The data in figure 9 were obtained in a similar manner for lead plate standards of 1.0, 1.9, and 4.6 mg/cm². The percentage a reading is reduced is relatively independent of the lead content of the sample, since 3 layers of ZnO-based paint reduce the reading for 76 mg/cm² by 13% and cause an 18% reduction in the reading for 4.6 mg/cm². Therefore, the reading obtained for a sample containing approximately 1 mg/cm² or less is almost independent of the effect of overlying layers of non-leaded paint. A 12% error occurs at the 1.0 mg/cm² level for either 14 overlayers of non-ZnO paint or 6 overlayers of 7% ZnO paint.

The manufacturer claims 88% penetration through 25 paint layers [9]. Experimentally, an 88% penetration was found to occur for 14 paint layers.

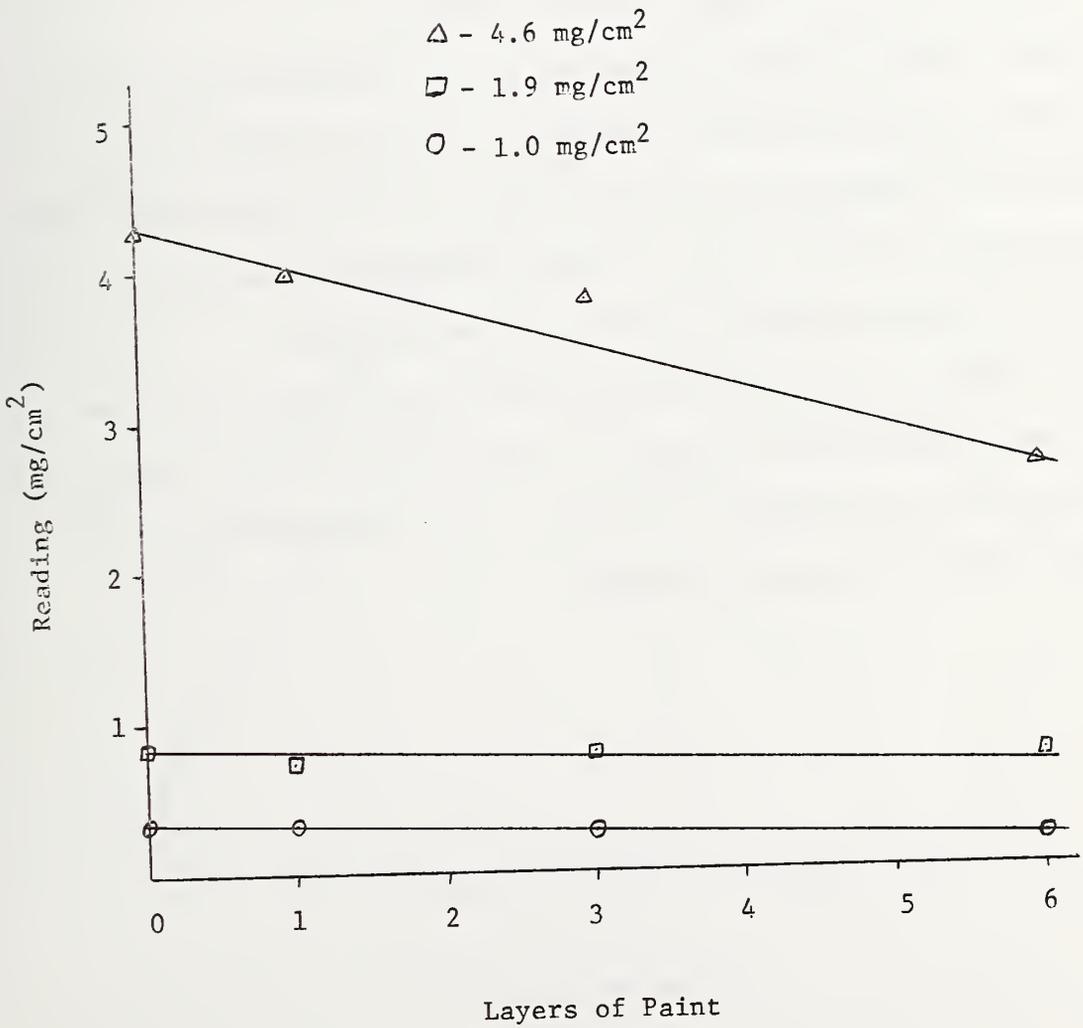
4.2.3. Substrate Effects

The substrate which supports the paint film causes a backscattering of some of the incoming x-rays. These scattered x-rays appear as noise which interferes with the analysis of the lead. The degree to which this occurs varies with the material and density of the substrate. The manufacturer claims that the variation in a reading caused by the substrate will not exceed 0.5 mg/cm² [7]. This claim seems to be substantiated by the results of a brief survey of substrate effects. However, an uncertainty of 0.5 mg/cm², combined with a standard deviation of 0.4 mg/cm², could result in a substantial error at the 1 mg/cm² level.

The substrates that were used in this study are: (1) 0.5 cm masonite, (2) 1.6 cm gypsum board, (3) 1.9 cm plywood, (4) a single coat of aluminum paint on 1.0 cm pine, (5) 0.6 cm asbestos board, and (6) 0.1 cm tin sheet metal. The effect of these substrates on lead plates of 0.1,

Figure 9

Attenuation of Response from ZnO Paint Overlayers



0.5, and 1.9 mg/cm² is shown in figure 10. There seems to be no effect from masonite nor gypsum board, and only a slight effect from plywood, aluminum paint, and asbestos. However, a substrate effect is definitely present for tin.

A separate calibration curve, figure 11, was constructed by placing the lead plate standards on the tin sheet metal. It is not a completely valid calibration, however, since the substrate was 0.5 cm behind the lead film. Therefore, the observed effect in the field should be somewhat larger. A calibration curve approximating figure 11 is more valid than figure 5 for correcting readings obtained on metal surfaces.

4.2.4. Effect of Distance

The K x-rays of lead, because they are of relatively low energy, are strongly absorbed by the air between the sample and the detector. Therefore, any separation between the detector and the sample will cause a reduction in the lead reading. This effect increases rapidly with distance and can be important for oddly shaped surfaces.

The affect of distance was determined by placing a 1.9 cm wooden block over a lead foil of 73 mg/cm². A separation of 1.9 cm caused a 92% reduction in the indicated lead value, and a separation of 3.8 cm caused a 99% reduction in the reading.

Figure 10

Effect of Substrate on Instrument Response

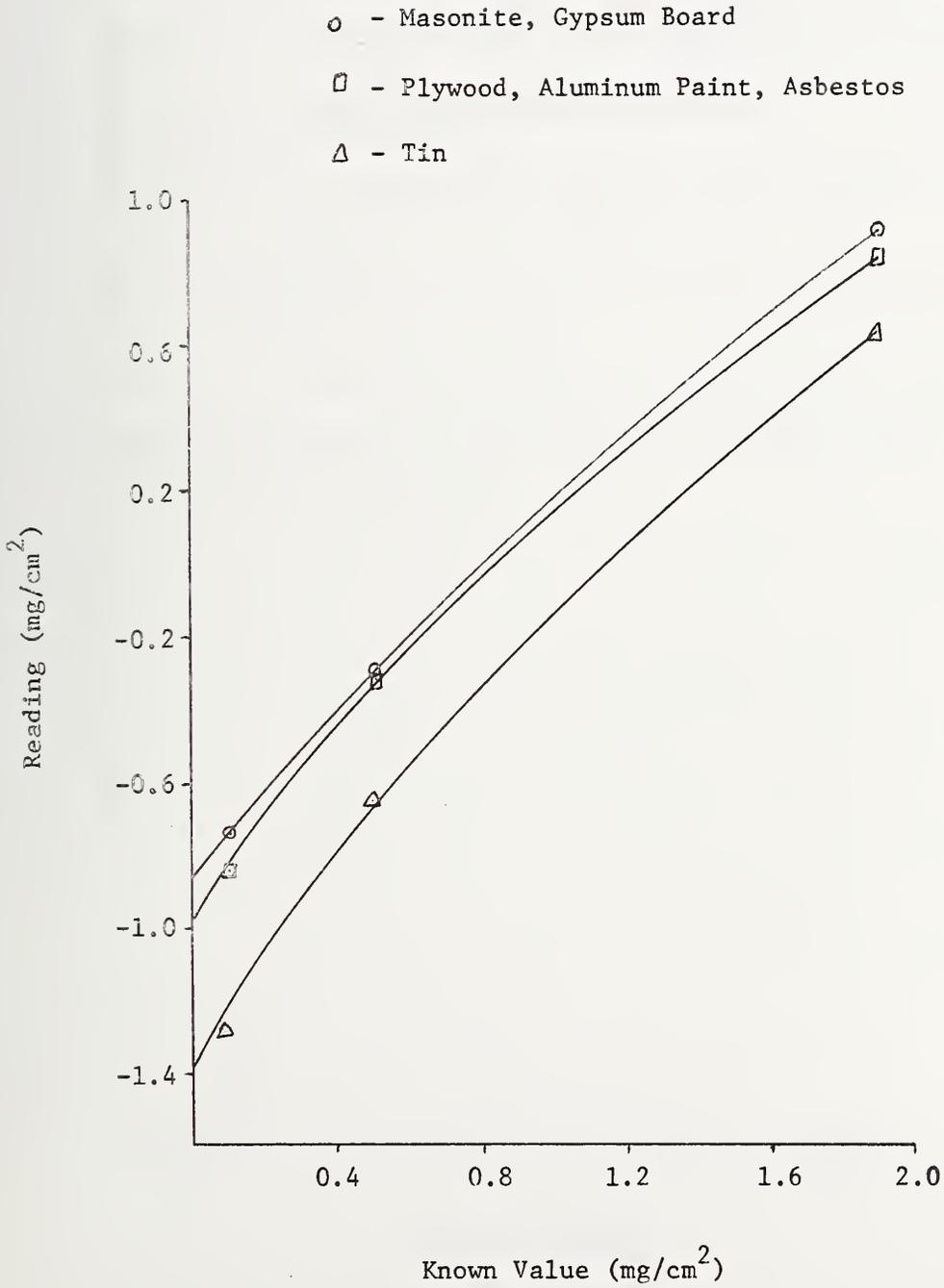
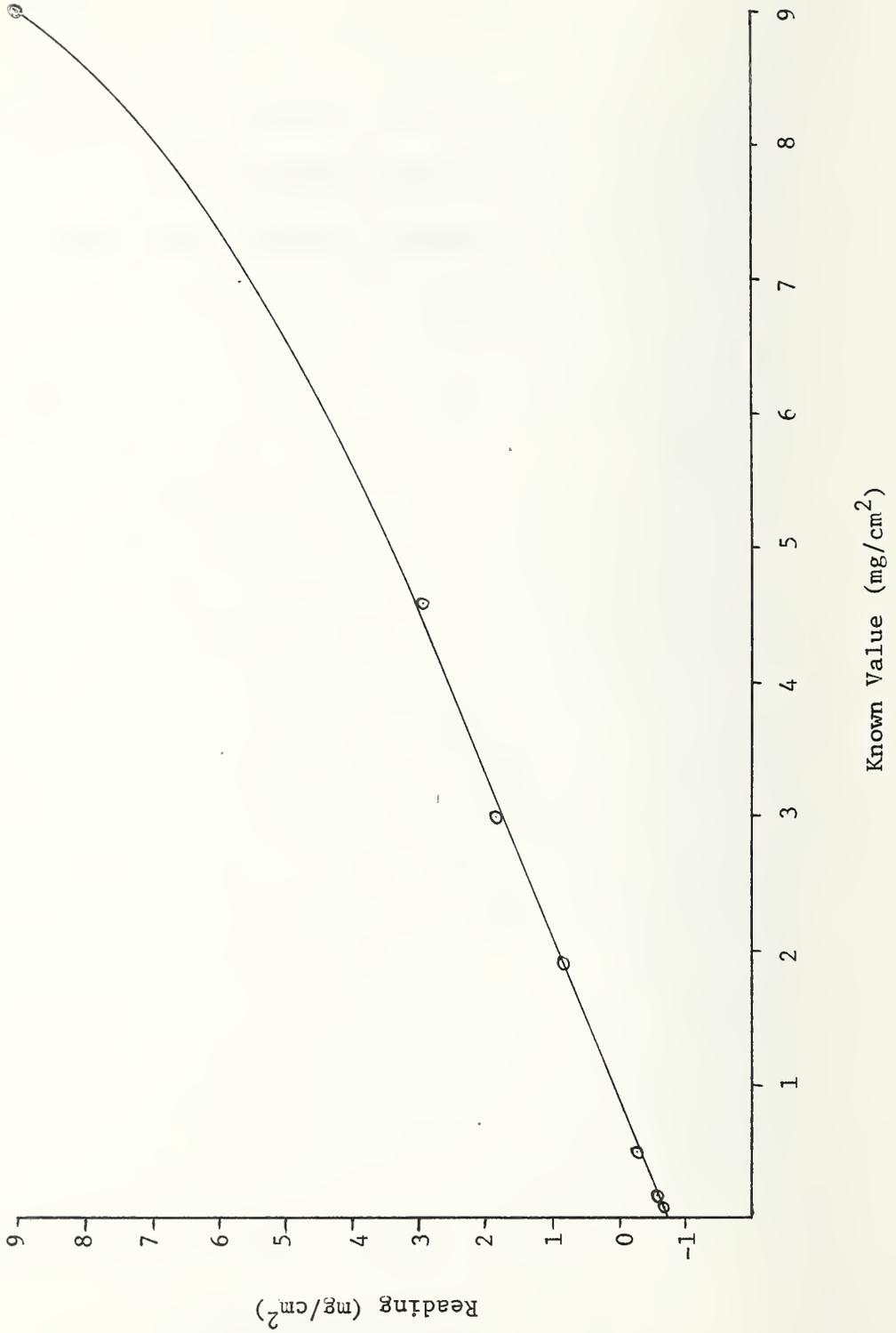


Figure 11

Calibration Curve for Metal Surfaces



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